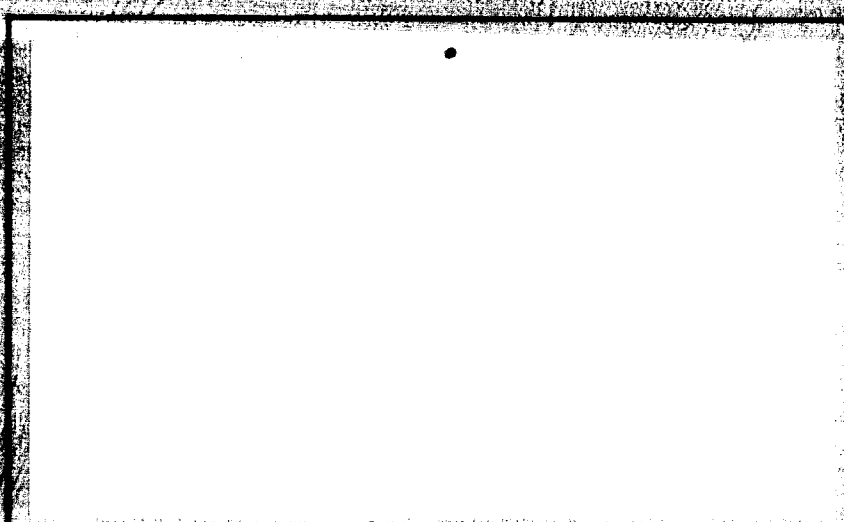


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SEVENTEENTH QUARTERLY PROGRESS REPORT

on

INVESTIGATION OF MECHANICAL PROPERTIES
OF CHROMIUM, CHROMIUM-RHENIUM,
AND DERIVED ALLOYS

to

NATIONAL AERONAUTICS AND SPACE
ADMINISTRATION

June 31, 1964

by

A. Gilbert and C. N. Reid

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505 King Avenue
Columbus, Ohio 43201

ABSTRACT

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The crystallography of slip and cleavage in Mo-35Re crystals has been studied during this report period. Twinning was completely suppressed, and slip on {112} planes was observed at 415 C and 28 C in a crystal with its longitudinal axis near to (001). Slip occurred on systems that were almost ideally oriented for the maximum shear stress.

Brittle cleavage fractures of similar crystals were performed by impact at a temperature near 80 K. The fracture propagated largely along {112}-type twin-matrix boundaries.

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INVESTIGATION OF MECHANICAL PROPERTIES OF CHROMIUM, CHROMIUM-RHENIUM, AND DERIVED ALLOYS

by

A. Gilbert and C. N. Reid

INTRODUCTION

The research carried out to date on the Metal Science Group Integrated Chromium Alloy Program has generally been oriented to investigate the value of three hypotheses put forward to explain the rhenium-alloying effect. A full discussion and evaluation of these hypotheses is included in the recent Yearly Progress Report^{(1)*}. In brief, these hypotheses have failed to provide all the answers as to why rhenium alloying is so effective in promoting ductility in chromium. In a new attempt to make headway in explaining the rhenium effect, work during the coming year will be devoted to a more intense investigation of the mechanical properties and deformation modes of ductile chromium-rhenium and chromium-ruthenium alloys.

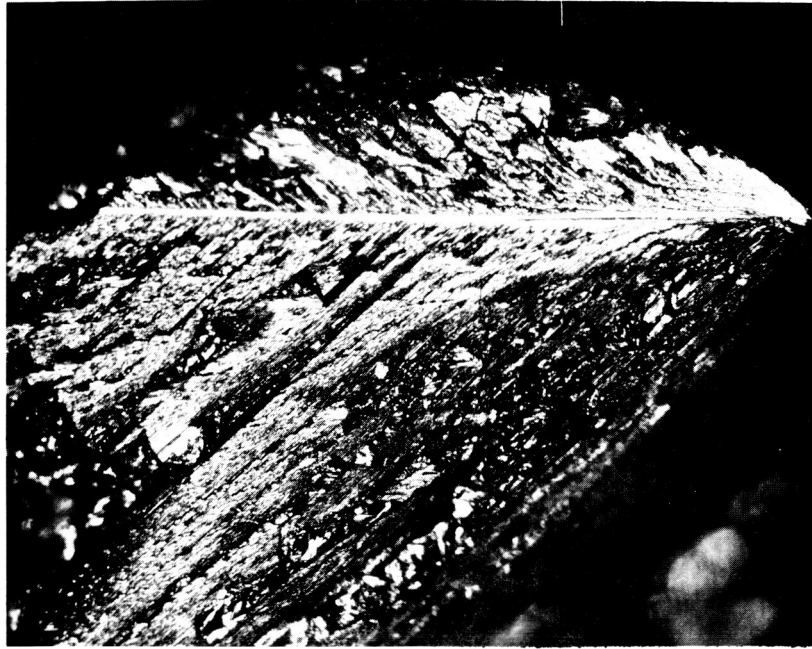
OBJECTIVES

As part of this program, it is intended to determine the identity of the active slip planes in high-rhenium alloys. For the sake of convenience, the investigations described in this report have been performed on Mo-35Re, because single crystals of this material can be produced readily. Ultimately, it is hoped to grow single crystals of Cr-35Re for the sake of a complete investigation, but until this has been accomplished, Mo-35Re can supply significant information.

In work recently completed on the Integrated Chromium Alloy Program, it was found that single crystals of chromium could fracture in a brittle manner at temperatures below -78 C.⁽²⁾ Figure 1 is a low-magnification photograph of the fracture surface of such a crystal fractured at -78 C. A pronounced line, observed on the fracture surfaces of brittle samples, was always found to lie along a $\langle 100 \rangle$ crystallographic direction. It was proposed that this line was the origin of fracture in these crystals, and arose from the intersection of two $\{110\}$ slip planes to produce a Cottrell crack. The action of $\{110\}$ slip planes in unalloyed chromium could thus lead to fracture initiation.

In order to see whether such a mechanism could operate in rhenium alloys, it was decided to determine the slip system and cleavage planes in a high-rhenium alloy. Fracture-surface markings similar to those in Figure 1 have also been observed in brittle failures of single crystal Mo⁽³⁾, an example of which is shown in Figure 2. Thus in view of the greater ease of single-crystal production in Mo-35Re, it was decided to study the slip mechanism and, if possible, the cleavage plane in Mo-35Re for comparison with those in unalloyed molybdenum.

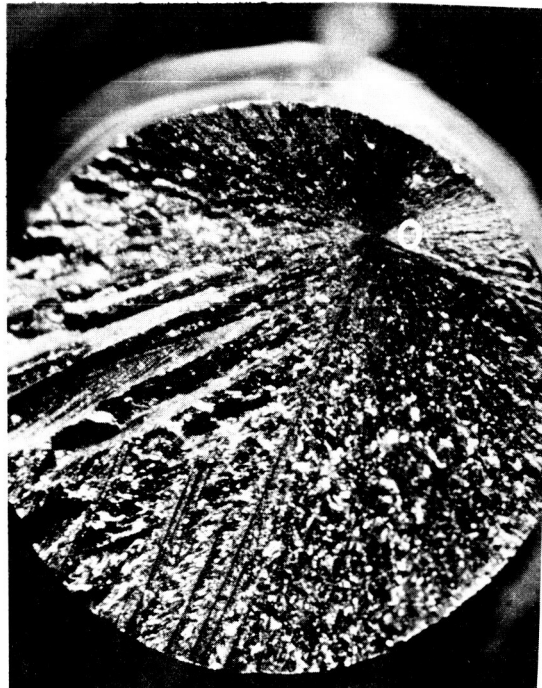
*References are given on page 9.



50X

7799

FIGURE 1. FRACTURE SURFACE OF CHROMIUM SINGLE CRYSTAL
THAT FAILED IN TENSION AT -78°C



20X

FIGURE 2. FRACTURE SURFACE OF MOLYBDENUM SINGLE CRYSTAL
THAT FAILED IN TENSION AT -196°C

EXPERIMENTAL PROGRAM

Slip Systems

In order to study the slip planes operative in Mo-35Re, it is necessary first to suppress twinning as a deformation mode, since the appearance of surface twins could easily make slip lines indistinguishable.

It has been found by several workers that deformation twinning occurs when the twinning systems are most highly stressed vis-à-vis the slip systems. (3, 4) Figure 3 illustrates that, for crystals loaded in compression, those with a longitudinal axis near to [001] are least favorably oriented for twinning, i. e., the applied load resolved onto the twinning systems is a minimum. This orientation of crystal then, should be the most reluctant to form twins in compression. Accordingly, such a crystal was grown, and compression cylinders, 0.160 inch in diameter and 0.50 inch in length, were prepared by grinding and electropolishing. The orientation of the crystal is also plotted in Figure 3. These samples were finally annealed in vacuum for 1 hour at 1500 C, followed by furnace cooling, in order to remove any work introduced by the grinding operation. The crystals did not recrystallize because the stored energy was small.

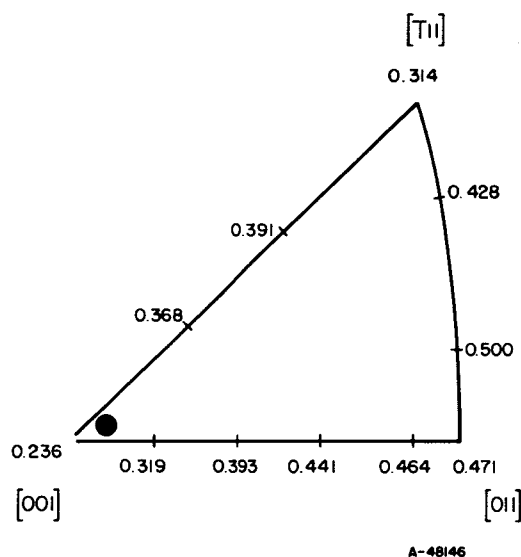


FIGURE 3. THE ORIENTATION DEPENDENCE OF SCHMID FACTORS FOR TWINNING IN COMPRESSION

The longitudinal axis of the crystals used is also plotted.

It is known that twinning may be suppressed by prestraining a specimen under conditions (e. g. high temperature) such that deformation proceeds by slip. (3, 5) Subsequently, the specimen will continue to deform by slip under conditions that normally produce twinning. This method was used to suppress twinning in addition to the choice of orientation and, thereby, the slip system operative at room temperature was observed.

A crystal was prestrained 2.1 per cent in compression at 415 ± 5 C at a rate of 0.4 per cent per minute, according to the stress-time curve shown in Figure 4. The slip produced by this prestrain was analyzed stereographically by plotting the angle between the specimen axis and the surface slip line at different points around the specimen's circumference. The slip plane determined in this way lay within 2 degrees of $(\bar{1}\bar{1}2)$. This system (assuming that the slip direction is $[111]$) carried a high shear stress, having a Schmid factor of 0.492. The maximum factor for a $\{110\}$ -type slip system was 0.448, so that slip occurred on the most highly stressed system. The sample was then electropolished to remove the surface slip markings and was compressed a further 2.1 per cent at room temperature (see Figure 4). The surface slip lines were again analyzed and found to represent the intersection of $(1\bar{1}2)$ planes with the surface, to within 5 degrees. Again, this system has a Schmid Factor of 0.492, and is the most highly stressed of the $\{110\}$ and $\{112\}$ -type systems.

In order to determine the effect of alloying with rhenium on the choice of slip system, it is necessary to know the operative slip plane in a Mo crystal of similar orientation, deformed under identical conditions. This is now in progress. A further experiment that suggests itself is to see if $\{110\}$ -type slip can be provoked at all in Mo-35 at. % Re by choosing a crystal orientation [with axis near $(\bar{1}23)$], such that a $\{110\}$ -type system is most highly stressed, and all the $\{112\}$ -systems are subjected to much smaller shear stresses. If it transpires that the alloy is reluctant to undergo $\{110\}$ -type slip, the repercussions on the theory of the rhenium-alloying effect would be great.

Cleavage Planes

Earlier studies of the tensile properties of high-rhenium alloys have revealed that these alloys do not normally undergo cleavage. Lawley and Maddin⁽⁶⁾ did observe one instance of cleavage in Mo-20 at. % Re, which took place on an anomalous $\{110\}$ -type plane; the normal cleavage surfaces in the bcc lattice are $\{100\}$ planes⁽⁷⁾. Recently, it was observed in this program that polycrystalline Cr-35 at. % Re fails partly by cleavage under impact loading.⁽¹⁾ Two types of facet were observed; the first kind was deduced from its markings to be a $\{112\}$ -type plane, perhaps a twin-matrix interface, while the second was termed "rhenium-modified" cleavage.

In order to positively identify these cleavage surfaces, it is necessary to work with material whose grain size exceeds the width of the X-ray beam, so that surfaces may be oriented by the Laue back-reflection method. So far, it has not been possible to grow single crystals of Cr-35 at. % Re, nor is it easy to produce grain sizes as large as 1 mm. Therefore it was decided to use single crystals of Mo-35 at. % Re again, for this study. Cr-35 at. % Re will be used at such time as single crystals become available.

A crystal of the orientation shown in Figure 3 was ground to cylindrical shape, 0.16 inch in diameter and 0.50 inch long, and a linear notch was introduced on one side, 0.010 inch in width and 0.040 in deep, with a radius of 0.005 inch. The samples were then annealed for 1 hour at 1500 C in order to eliminate the residual work due to grinding. Two specimens were then broken by impact loading in a Tinius Olsen machine having a capacity of 16.7 ft-lb and an impact velocity 11.4 ft/sec. Specimens were mounted vertically in an adapter block, with the notch facing the pendulum. The block and specimen were cooled by immersion in liquid nitrogen and were swiftly transferred

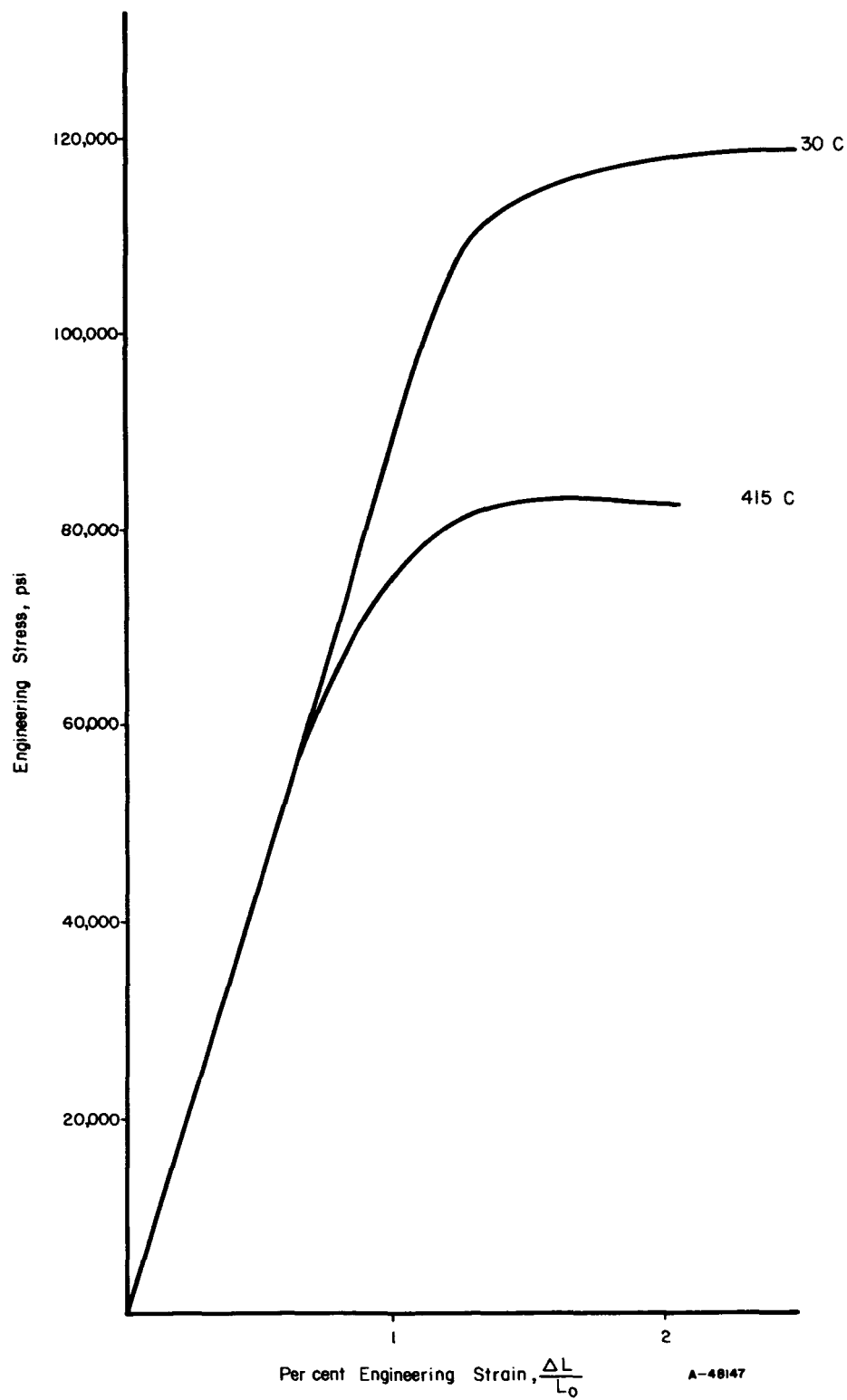


FIGURE 4. STRESS-STRAIN CURVES FOR SINGLE CRYSTAL OF Mo-35Re TESTED IN COMPRESSION AT 415 C AND 30 C

to the machine and impacted within about 10 seconds. Since the object was to induce a cleavage rather than to determine the energy absorbed at 78 K, any warming of the specimen above 78 K that may have occurred is of no consequence. However, for the record, Crystal 2 absorbed 11 in-lb, and Crystal 3 absorbed 38 in-lb in undergoing complete fracture.

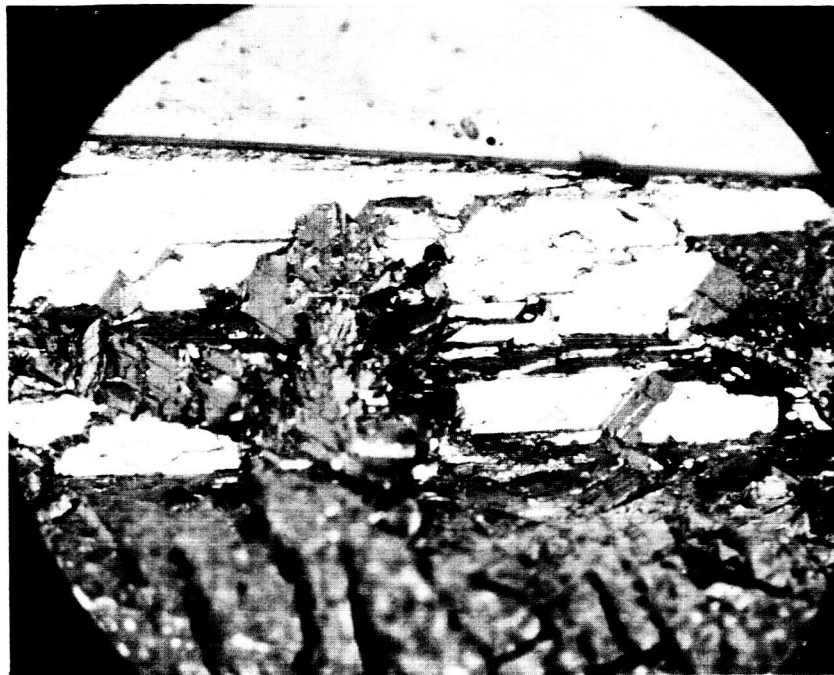
The failure propagated from the root of the notch, and contained two types of surface, as illustrated in Figure 5. The first type occupied the half of the cross section adjacent to the notch and consisted of large flat facets such as that shown at higher magnification in Figure 6. When two such facets were aligned under a microscope and a Lauegram taken, the plane of the surface was positively identified as a $\{112\}$. Their appearance is very similar to those illustrated in the last Yearly Progress Report⁽¹⁾ from impacted Cr-35 Re. Longitudinal sectioning revealed that these $\{112\}$ facets are in fact the boundaries of mechanical twins, as may be seen in Figure 7, where the fracture surface alternates to either side of the twin at the surface.

The second type of surface was located on the part of the cross section opposite to the notch. This surface consisted of many small facets, which did not readily lend themselves to photography due to their uneven surfaces. However, longitudinal sectioning revealed that these facets also frequently consist of twin boundaries. Their small size seems to be related to the fact that the network of twins is more closely packed in this part of the specimen. The twins have also suffered much deformation by slip and are frequently curved in shape; this may account for the roughness of the facets.

In summary then, the path of crack propagation was predominantly along twin boundaries. No preference for another type of plane was observed, although the $\{112\}$ facets were sometimes linked by short sections of unidentified and confused trans-crystalline crack. Previous research has showed that when grain boundaries are present, they provide preferential sites for crack propagation, and now in their absence, it is found that mechanical twins form under the stress of impact, and thereby provide a particular kind of grain boundary for the crack to follow (a twin boundary is, in essence, an interface between two grains that are twin related). This behavior is in striking contrast to Molybdenum⁽³⁾ and Iron⁽⁸⁾ with which the formation of twin intersections leads to cleavage on $\{100\}$ planes rather than parting of the twin boundary. Although the crystals above were ideally oriented for crack propagation along (001), this did not occur. The reason for this reluctance to undergo $\{100\}$ cleavage is not known at present. It was suggested above that it may reflect a difficulty in forming the Cottrell dislocation association mechanism, due to the absence of slip on $\{110\}$ -type planes. The current observation of slip planes will test this. So far, it has been shown that slip can occur in Mo-35 at. % Re on $\{112\}$ planes, when the planes are almost ideally oriented, but it has not yet been determined whether slip will occur in a crystal oriented for $\{110\}$ -type slip. When this is done, the above theory will be put to the test.

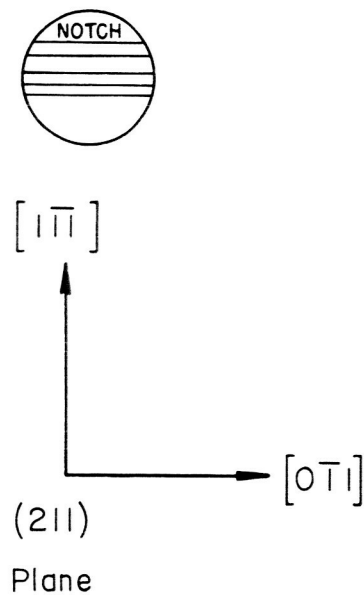
Failure along twin-matrix boundaries is not unprecedented; Berry⁽⁹⁾ observed similar behavior in Fe-3.5%Si, as did Low⁽¹⁰⁾ in unalloyed iron.

It would be interesting to know the mechanism of failure of a Mo-35 at. % Re crystal in the absence of twins and grain boundaries. Would $\{100\}$ cleavage then occur? It is intended to find this out by impacting a crystal in the above manner, after pre-straining by slip. This experiment should indicate whether in high-rhenium alloys $\{100\}$ cleavage has become more difficult, or cleavage along twin boundaries has become easier. This, and the experiments on slip systems referred to above, will continue into the next report period.



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FIGURE 5. MACROGRAPH OF FRACTURE SURFACE OF Mo-35 Re SINGLE CRYSTAL BROKEN BY IMPACT AT APPROXIMATELY -196°C

The $[0\bar{1}1]$ direction marked is the line of intersection of (211) and $(2\bar{1}\bar{1})$

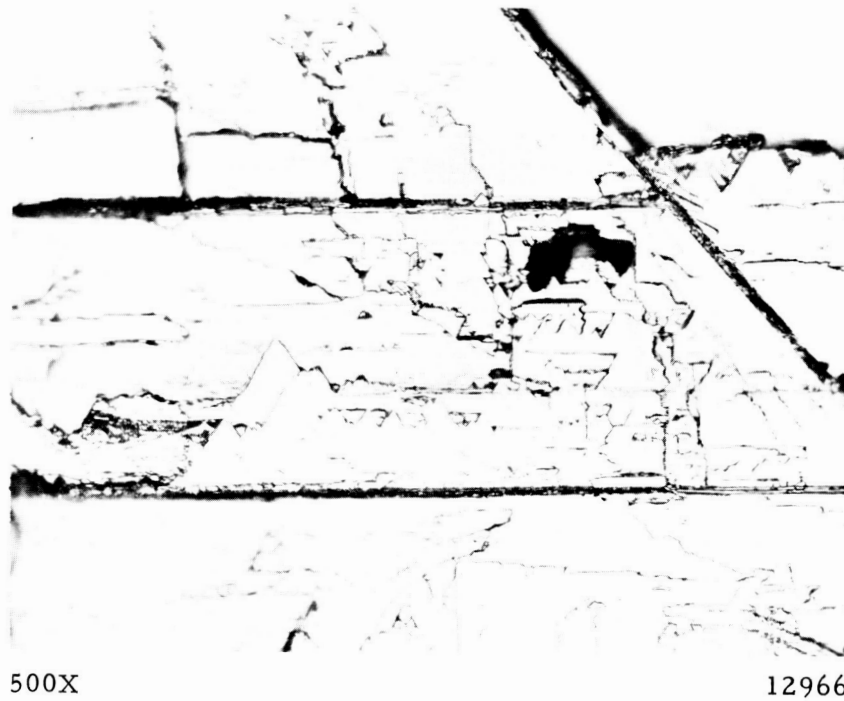


FIGURE 6. FRACTOGRAPH OF PART OF THE LIGHT AREA SHOWN IN FIGURE 5 SHOWING DETAILED MARKINGS



FIGURE 7. METALLOGRAPHIC SECTION THROUGH FRACTURE SURFACE OF Mo-35 Re SINGLE CRYSTAL

The light area at the lower left is nickel plating.

SUMMARY

- (1) Twinning at room temperature may be suppressed in a [100] single crystal of Mo-35 at. % Re by prestraining at 415 C.
- (2) Slip in this crystal occurred on $\{112\}$ planes at both 415 C and 28 C.
- (3) A crystal of this orientation will undergo brittle fracture under impact at a temperature in the vicinity of 78 K.
- (4) The fracture propagates largely along $\{112\}$ twin boundaries, under these conditions.
- (5) No $\{100\}$ cleavage was observed.

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